

Operations and Maintenance Manual for the Water Reclamation Plant (WRP)

Prepared for

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APPENDIX

A Water Discharge Regulations	
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18. SAMPLING AND LABORATORY PROCEDURES

18.1 INTRODUCTION

This section outlines influent, effluent, and process parameter sampling techniques and laboratory analytical procedures critical to:

- Provide data essential for facility control and maximization of operational efficiency.
- Maintain historical records on facility performance.
- Verification of EPA Rule Authorization compliance.

There are two good references to have at the WRP for details on all sampling procedures and handling: *Standard Methods for the Examination of Water and Wastewater* and WEF's *Wastewater Sampling for Process and Quality Control* (MOP OM-1).

18.1.1 Sampling Schedule

Tables 18-1 and 18-2 summarize recommended facility testing requirements for influent and effluent, respectively. Table 18-3 summarizes facility testing requirements to verify that effluent is meeting the requirements of the EPA Rule Authorization further described in Chapter 2.

Table 18-1. Influent Sampling Schedule

Parameter	Units	Minimum Sampling Frequency	Sample Type
Flow	mgd	Continuous ^a	Recording meter
BOD5	mg/L lbs/day	Weekly	24-hour composite
TSS	mg/L lbs/day	Weekly	24-hour composite
pH	Standard Units	Daily	Grab

^a Continuous means uninterrupted except for brief lengths of time for calibration, for power failure, or for unanticipated equipment repair or maintenance. Sampling shall be taken every 4 hours when continuous monitoring is not possible.

Table 18-2. Effluent Sampling Schedule

Parameter	Units	Sampling Point ^a	Minimum Sampling Frequency	Sample Type
Flow	mgd	Disinfected Permeate Water	Continuous	Recording meter
BOD5	mg/L	Disinfected Permeate Water	Weekly	24-hour composite
BOD5	lbs/day	Disinfected Permeate Water	Weekly	Calculation
BOD5	% removal	Disinfected Permeate Water	Weekly	Calculation
TSS	mg/L	Disinfected Permeate Water	5/week	24-hour composite
TSS	lbs/day	Disinfected Permeate Water	Weekly	Calculation
TSS	% removal	Disinfected Permeate Water	Weekly	Calculation
pH	Standard Units	Disinfected Permeate Water	Daily	Grab ^b

(Table Continues)

Table 18-2. Effluent Sampling Schedule (Continued)

Parameter	Units	Sampling Point ^a	Minimum Sampling Frequency	Sample Type
Temperature	Celsius	Disinfected Permeate Water	Daily	Grab ^b
Turbidity	NTU	Permeate Water before Disinfection	Continuous ^c	Recording meter
Total Nitrogen (as N)	mg/L	Disinfected Permeate Water	Monthly	24-hour composite
Ammonia (as N)	mg/L	Disinfected Permeate Water	Weekly	24-hour composite
Nitrate (as N)	mg/L		Weekly	24-hour composite
Total Coliform ^d	No. of org. per 100 ml	Disinfected Permeate Water	2/Week	Grab ^b
Free Chlorine Residual	mg/L	Plant Reclaimed Water	Daily (when in use)	Grab ^b

^a Disinfected permeate water samples shall be taken from 8-inch RCL pipe.

^b Grab samples shall be taken at the same time daily when wastewater characteristics are the most demanding on the treatment facilities and disinfection processes.

^c Effluent turbidity analysis shall be performed by a continuous recording turbidimeter and shall also be read and recorded at least once per day.

^d As an alternate method, total coliform bacteria may be monitored using the ONPUG-MUG test (also called Autoanalysis Colilert System)

18-3. EPA Rule Authorization Sampling Schedule

Parameter	Units	Sampling Point ^a	Minimum Sampling Frequency	Sample Type
Primary Drinking Water Standard Listed in Appendix A	See Appendix A	Post UV Disinfection	Quarterly first year of operation Semiannual following first year	24-hour composite
Primary Drinking Water Standard Listed in Appendix A	See Appendix A	Monitoring Well, Injection Site	Semiannual first 2 years of operation Annual following second year.	24-hour composite
Primary Drinking Water Standard Listed in Appendix A	See Appendix A	Monitoring Well, North Edge of Property	Annual following third year of operation	24-hour composite
Free Chlorine Residual	mg/L	Post Reclaimed Water Storage Tank	Daily (when in use)	Grab ^a
Free Chlorine Residual	mg/L	Active Monitoring Well(s)	Weekly (when in use)	Grab ^a

^a Grab samples shall be taken at the same time daily when wastewater characteristics are the most demanding on the treatment facilities and disinfection processes.

18.1.2 Process Control Monitoring for Membrane System

The MBR system and individual system components are discussed in detail in Chapter 10. Operational monitoring protocols are required to determine the efficiency of the MBR treatment system and to control specific loading conditions.

Table 18-4 is a partial list of suggested parameters and sampling frequency recommended in the O&M manual that was provided by Kubota Membranes of America, Inc. (Kubota Table 1.3-2).

Table 18-4. MBR Recommended Operational Monitoring Requirements

Parameter ^a	Required Monitoring Frequency ^a	Sample Location ^b IN/EFF/MBR ^c
Permeate Flow	1/min	EFF
Air Scour Flow	1/min	MBR
Mixed Liquor TSS	2/week	MBR
Water Temperature	1/day	MBR
TMP	1/min	MBR
pH	2/week	IN/EFF/MBR
DO	1/day	MBR
Filterability	1/day	MBR
Turbidity	1/week	EFF
NH3-N	1/week	IN/EFF

^a Parameter type and testing frequency assume typical municipal waste. For industrial applications or other, requirements can change.

^b IN/EFF/MBR refer respectively to; influent wastewater downstream of screening, permeate downstream of SMUs only (not post disinfection), and mixed liquor inside the MBR.

^c Collect 24-hour composite samples for influent and effluent testing. For mixed liquor lab samples, simply submerge a container directly into the MBR. Follow sampling protocol as required for lab analysis of DO.

It would also greatly benefit the operators to maintain a history of influent nitrogen loads to the facility (TN, TKN, nitrates, etc.) to evaluate nitrogen removal and remaining removal capacity of the system.

18.1.3 Sampling Procedures

The following steps should be followed by the treatment plant staff to obtain quality and representative samples:

- Samples collected on a routine basis should be taken from the same location at the same time of day or week using the same procedures.
- Samples should be taken at locations where the wastewater is as completely mixed as possible.
- When sampling, particles larger than 1/4 inch, floating materials and debris should be excluded, or removed.
- In situations where the interval between sample collection and analysis is long enough to produce changes in concentration or physical states of constituents to be analyzed, preservation techniques such as refrigeration or immersion in ice water should be utilized.

- All sampling equipment such as bottles, automatic samplers, and other collection devices should be properly cleaned and maintained.
- When testing influent and effluent samples, the relationship between the plant's flow variation and detention time should be considered so that analyses are performed on samples taken from the same waste. This primarily applies to grab samples.
- When utilizing automatic samplers, make sure that sampling personnel understand intake placement, power requirements, and timer/volume adjustments.
- No samples should be taken when there are unusual return flow conditions (i.e., dewatering of a structure) unless the operator is specifically trying to determine the effect of the recycle stream.
- Make sure the automatic sampler's purge cycle is of sufficient duration to completely evacuate the intake line before each sampling cycle.

18.1.4 Sample Handling

Collect enough samples in a suitable container. Error can result from attempting to collect small portions for a composite sample. A minimum sample should be at least 100 ml and a sample containing any unusual particles should be rejected.

Always mix the sample before removing a sub-sample for analysis. If this operation is neglected, the liquid removed will not represent the original. Mixing may be accomplished by shaking, stirring, or other means, depending on the analysis to be conducted and the judgment of the analyst. Careful stirring to avoid aerating the sample is suitable for a BOD test, whereas shaking is fine for a solids determination.

When removing a measured portion of sample, use the appropriate method. For example, if a portion of 50 or 100 ml is desired for a test such as suspended solids, use a graduated cylinder or volumetric flask. The sample should be thoroughly mixed and rapidly poured into the measuring device. It is preferable to miss the exact amount by a small degree rather than to use extreme care in pouring. Extreme care in pouring allows time for the particles in suspension to settle, resulting in error. In a test, such as a total solids or suspended solids in wastewater, the cylinder or flask can be rinsed with distilled water and the rinsings added to the evaporating dish or crucible without adversely affecting the results.

Smaller portions which might be used for BOD tests can be measured with a pipette. However, a fine-tipped pipette can cause error by straining out larger particles. If the opening is sufficiently large, the pipette can be filled by dipping into the container of mixed sample, eliminating the need for using a pipette bulb. Only one portion should be pipetted at a time, as the delay in dispensing more than one portion from the same pipette allows the contents to separate in the pipette.

Preserve the sample during any delay before analysis. For routine wastewater tests, refrigeration is adequate. The temperature should be 4 degrees C (30 degrees F). With a thermometer inserted through the stopper of a refrigerated bottle of water, the temperature can be read without the rapid change that results from opening a refrigerator and reading an unprotected thermometer.

18.1.5 Sampling Methods

There are two different types of sampling methods. One method is grab sampling, which consists of a single sample generally taken randomly at no set time or flow. The other is a composite sample which is made up of smaller samples collected at set times or flows during a given period. Both grab and composite samples can be collected either by hand or automated

sampler, although composite samples are more representative when collected via an automated sampler.

Composite or integrated samples are collected when a measure of the average quality or condition in the plant streams is sought. Composite sampling covers a period usually no longer than 24 hours and is commonly conducted for routine analyses of solids, BOD, etc. Ideally, a continuous sample should be taken with volumes at all times in proportion to rate of flow for the waste stream of interest. This capability is provided by automatic influent and effluent samplers. Where this is not practical, samples taken hourly and composited may give reasonably accurate results. Greater frequency may be required where sudden changes occur causing wide variations in wastewater composition and flow.

Where samples are collected during a period less than 24 hours (during an operator shift), a relationship between the results found during the shorter period and the daily average should be established. A factor representing this relationship should thereafter be applied to compute daily volumes.

Satisfactory results are also obtained by collecting a portion of uniform size each time a predetermined amount of flow has passed the sampling station. Using this scheme, samples are collected more often during high flows and less often during low flows.

18.1.6 Sampling Equipment

18.1.6.1 Sampling Containers

For grab sampling, well-cleaned and rinsed large-mouth glass or plastic containers should be used. It is important that these containers are used for sampling purposes only.

All sampling containers should be well cleaned, then rinsed with the waste being sampled several times. It should be noted that the fecal coliform test requires sterilized containers to preclude the possibility of background contamination of samples. Sterilization can be accomplished by either heating glass containers without their lid in an oven at 350 degrees F (121 degrees C) for 2 hours or by the use of the autoclave (use only glass containers for fecal coliform sampling or autoclaved plastic containers). After the containers have been sterilized, the lids must be put back on tightly as soon as the glass containers and lids are removed from the oven and allowed to cool slightly.

18.1.7 Sample Preservation

All samples must be properly preserved according to the analysis to be performed. Failure to treat samples properly will result in errors. Preservation techniques retard chemical and biological changes, which occur after sample collection. Methods of preservation are intended to:

- Retard biological growth.
- Retard hydrolysis of chemical compounds and complexes.
- Reduce the volatile nature of some chemical constituents.
- Reduce absorption effects, such as ion exchange between complexes.

Preservation methods are generally limited to pH control, chemical fixing and refrigeration or freezing. Refrigeration at a temperature of 3 to 4 degrees C has been shown to be the most effective method of preservation for both BOD and TSS samples. Dissolved oxygen, Cl₂ residual, and pH samples should be prepared and analyzed in-situ or immediately after sample collection. Dissolved oxygen measurements are particularly sensitive to temperature

fluctuations and should be conducted at the source. Dissolved oxygen solubility decreases with increasing temperature; therefore, a DO sample analyzed in a relatively warm lab would tend to underestimate DO concentration in the original sample.

18.1.8 Sample Recording

It is important that an accurate record be kept for each sample collected, and that every bottle is clearly labeled and tracked. Sampling records should contain the following where applicable:

- Name of the sample collector.
- Date and time of sample collection.
- Name of the sample analyzer.
- Date and time of sample analysis.
- Type(s) of analysis conducted.
- The exact location and nature of the sample.
- Water temperature (if applicable).
- Other applicable data such as flow at time of sampling.
- Weather conditions.
- Preservation technique, if applicable.
- Analytical technique or method.

Maintenance of accurate daily records used in the operation of the treatment facility will provide the framework for the required monthly reports. Good records are also necessary for process troubleshooting. In addition, it is recommended that weather conditions such as temperature, rainfall, and other hydrological data, along with wastewater physical characteristics such as temperature and pH, be recorded on a daily basis.

18.1.9 Record Keeping

It is recommended that the tribe retain records of all monitoring information for a minimum of 3 years. Such information must include all calibration and maintenance records and all original recordings for continuous monitoring instrumentation, copies of all reports, and records of all data. All laboratory reports providing data for organic and metal parameters must include the following information: sampling date, sample location, date of analysis, parameter name, CAS number, analytical method/number, method detection limit (MDL), laboratory practical quantitation limit (PQL), reporting units, and concentration detected. Analytical results from samples sent to a contract laboratory must include information on the chain of custody, the analytical method, QA/QC results, and documentation of accreditation for the parameter.

OPERATOR'S NOTE: It is the recommendation of this manual to collect and store all data in an electronic database indefinitely. This will serve as a reference that could help diagnose process-related issues and educate new operators on the historic function of the facility under various conditions.